

ICS 83.080.01



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 19466.3-2025

**Plastics - Differential scanning calorimetry(DSC) method - Part
3: Determination of temperature and enthalpy of melting and
crystallization**

(English Translation)

Issued on: December 2, 2025

Implemented on: July 1, 2026

Issued by: State Administration for Market Regulation

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1 Scope

This document describes a test method for determining the temperature and enthalpy of melting and crystallization of crystalline and semi-crystalline polymers using differential scanning calorimetry (DSC) method.

This document applies to the determination of temperature and enthalpy of melting and crystallization of crystalline and semi-crystalline polymers.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 2035 Plastics - Vocabulary (GB/T 2035-2024, ISO 472:2013, NEQ)

GB/T 19466.1 Plastics - Differential scanning calorimetry (DSC) method - Part 1.

General principles (GB/T 19466.1-2025, ISO 11357-1:2023, MOD)

3 Terms and definitions

For the purpose of this document, the terms and definitions defined in GB/T 2035 and GB/T 19466.1, as well as the following apply.

3.1

melting

Process by which a fully crystalline or semi-crystalline polymer transforms from a solid state to an amorphous liquid state with different viscosities.

NOTE: Melting is also called fusion, and it appears as an endothermic peak in the DSC curve. For liquid crystal polymers, "amorphous liquid state" is replaced by "ordered liquid state".

3.2

crystallization

Process by which a polymer transforms from an amorphous liquid state to a fully crystalline or semi-crystalline solid state.

NOTE: Crystallization appears as an exothermic peak in the DSC curve. For liquid crystal polymers, "amorphous liquid state" is replaced by "ordered liquid state".

3.3

enthalpy of fusion

Amount of heat absorbed by a material during melting at constant pressure.

NOTE: In kilojoules per kilogram (kJ/kg) or joules per gram (J/g).

3.4

enthalpy of crystallization

Heat released by the crystallization of a material at constant pressure.

NOTE: In kilojoules per kilogram (kJ/kg) or joules per gram (J/g).

4 Principle

See the provisions of GB/T 19466.1.

5 Instruments and materials

It shall comply with the provisions of GB/T 19466.1.

6 Specimen

It shall comply with the provisions of GB/T 19466.1.

7 Test conditions and specimen conditioning

It shall comply with the provisions of GB/T 19466.1.

8 Calibration

It shall comply with the provisions of GB/T 19466.1.

9.1 Instrument preparation

It shall comply with the provisions of GB/T 19466.1.

9.2 Specimen loading

Specimen loading shall comply with the provisions of GB/T 19466.1.

Place the specimen in a crucible, weigh the specimen, to an accuracy of 0.1 mg. Unless otherwise specified in the material standard, the preferred specimen size is 5 mg ~ 10 mg. However, when the heat of transition is high or low, the required specimen mass may exceed 5 mg ~ 10 mg. For polymer compounds, considering the actual composition of the polymer, the mass used shall be the mass of the matrix polymer.

9.3 Crucible loading

It shall comply with the provisions of GB/T 19466.1.

9.4 Testing

9.4.1

Before starting the heating cycle, purge with nitrogen for 5 min.

9.4.2

It should preferably start heating at a rate of 10 K/min or 20 K/min and record, but a higher heating rate may be used to eliminate the thermal history of the specimen during the first heating.

9.4.3 Perform and record the first heating procedure, heating the crucible to a

sufficiently high temperature to eliminate the thermal history of the specimen, typically about 30 °C above the extrapolated end melting temperature ($T_{ef,m}$).

The thermal history and morphology of the specimen significantly affect the test results of

the polymer's melting and crystallization temperatures and enthalpies, so it shall perform a preheating cycle and a second heating procedure (see GB/T 19466.1-2025).

If the material is reactive or it is desirable to assess the effect of a single heating cycle on the specimen's properties, the data from the first thermal cycle shall be used as the test results. The test report shall record the differences from the standard procedure, including the number of cycles.

9.4.4 Hold the temperature for 5 min.

NOTE: If the polymer has not degraded, a longer holding time may be used.

9.4.5 It should preferably perform cooling at a rate of 10 K/min or 20 K/min and record, cooling to about 50 °C below the extrapolated end crystallization temperature ($T_{ef,c}$).

When simultaneously determining the polymer's glass transition and crystallization process, it shall cool to 50 °C or lower below the glass transition temperature.

NOTE 1: In addition to the heating and cooling rates recommended in this document, other heating or cooling rates may be used upon agreement between the relevant parties. High heating and cooling rates improve test sensitivity, while low heating and cooling rates provide better resolution.

Choosing appropriate heating and cooling rates is crucial for observing subtle changes.

NOTE 2: Due to supercooling, crystallization only occurs when a certain temperature gradient is reached, typically below the melting temperature.

9.4.6 Hold the temperature for 5 min.

NOTE: Limiting the thermal history is important for accurately evaluating test results.

9.4.7 Perform a second heating to approximately 30 °C above the extrapolated end

melting temperature $T_{ef,m}$ and record. The heating rate should ideally be the same as the cooling rate (see 9.4.5).

9.4.8 Cool the instrument to room temperature, remove the crucible, and check for

crucible deformation or specimen overflow.

9.4.9 Reweigh the crucible and specimen unless it is certain that there was no mass loss during the test. If significant mass loss occurs, remeasure using a fully dried specimen.

10.1 Calculation of transition temperatures

Adjust the coordinate axis range so that the peak coverage reaches more than 25 % of the vertical axis. Draw a baseline by connecting the initial temperature $T_{i,m}$ and the end